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CHARACTERIZATION OF LUNAR ILMENITE RESOURCES

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Abstract

Ilmenite will be an important lunar resource, to be used mainly for oxygen production but also as a source of iron. Ilmenite abundances in high-Ti basaltic lavas are higher (10-20%) than in high-Ti mare soils (mostly <10%). This factor alone may make crushed High-Ti basaltic lavas most attractive as a target for ilmenite extraction. Concentration of ilmenite from either a crushed basalt or regolith requires sizing to avoid polycrystalline fragments. In coarse-grained high-Ti basaltic lavas, about 60-80% of the ilmenite will consist of relatively "clean" single crystals if the rocks are crushed to a size of 0.2 mm. Fine-grained high-Ti basalts , with thin skeletal or hopper-shaped ilmenites, would produce essentially no free or "clean" ilmenite grains unless crushed to sizes of less than 0.15 mm, and only ~7% free ilmenite if crushed to sizes smaller than 0.05 mm . Data from the 2.8 m-thick regolith sampled by coring at the Apollo 17 site show that in even the most basalt-clast-rich and least mature stratigraphic intervals, free ilmenite grains make up less than 2% of the 0.02- to 0.2-mm size fraction and a mere 0.3% of the 0.2- to 2 mm size fraction.

Introduction

The high-titanium mare basaltic lavas and pyroclastic deposits, which are rich in ilmenite ($FeTiO_3$), chromite ($FeCr_2O_4$), and troilite (FeS), are important to the development of self-sufficient lunar colonies; Ilmenite is the major resource for oxygen; other products that may be obtained from these three minerals include sulfur, iron, and chromium. Vaniman et al. (in press) outline potential uses for sulfur, abundant in high-Ti lunar basalts (0.16—0.27% by weight), which will be an important byproduct of oxygen production.

On the lunar nearside, the maria cover an area of 6.4×10^6 km^2, which is about 17% of the Moon's surface (Head, 1976). The total volume of lunar basaltic lavas and associated pyroclastic rocks is estimated at between 10^6 and 10^7 km^3 (dependant on models used for thicknesses of basalt in the ring-basins). High-Ti lavas and pyroclastic materials make up approximately 20% of the visible mare units (BVSP, 1981). Spectral data and samples collected during the Apollo 11 and 17 missions indicate that the Tranquillitatis and Procellarum basins in particular contain abundant near-surface high-Ti basalts (Pieters, 1978). High-Ti basalts are thus a vast resource, with a minimum volume of 2×10^4 km^3.

McKay and Williams (1979) determined that regolith developed on high-Ti basalts may have as much as 5% ilmenite that is readily available as a resource. This is, however, for an immature soil with little or no exposure at the lunar surface. By comparison, much higher ilmenite contents (9-19%) are locked in the high-Ti basaltic rocks.

Beneficiation of ilmenites will depend on the volume of single ilmenite crystals that can be extracted with the least amount of energy needed to crush the source material. The efficiency of this operation will depend upon: (1) the grain size of ilmenite, (2) the grain shape, and (3) the degree to which the rock serving as feedstock has already been comminuted by brittle fracture of cooling and solidifying flow surfaces and by the breakup of lava flows by ongoing impact processes at the lunar surface.

The purpose of this study is to quantitatively characterize the sizes and shapes of ilmenite crystals within a variety of lunar samples collected at the Apollo 17 and Apollo 11 sites (located on high-Ti maria) and to evaluate their utility as feedstock for oxygen production. Basic descriptions of ilmenite shapes, volumes, and chemical compositions appeared in the earlier Lunar Science Conference Proceedings, when investigators were getting their first looks at the lunar rocks (e.g., Brown et al., 1970; Cameron, 1970; El Goresy et al., 1974). During later missions and after the end of the Apollo Program, very few basic, systematic rock descriptions were published; exceptions to this lack of descriptive data include the reports by Warner et al. (1976, 1978), who catalogued the Apollo 17 basalt samples that were collected by raking lithic clasts from the regolith.

For this study, we have reviewed the data on ilmenites for 109 samples from the Apollo 11 and Apollo 17 landing sites, including lavas, regolith breccias, and pyroclastic deposits. Using polished thin sections, we collected size, shape, and chemical data for ilmenites from 30 of these samples.

Analytical Procedure

For this work, we have used polished thin sections in a scanning electron microscope. After acquiring a high-contrast backscattered electron image, the image is processed, using a Tracor Northern Vista® image processing system.

To process the image, gray levels are segmented to separate the oxide minerals from iron and troilite, with high average atomic numbers per unit volume, and from the common silicate minerals, with low average atomic numbers per unit volume. In developing our identification technique, identifications were verified with x-ray spectra and electron microprobe analyses. A binary image was constructed from the segmented gray level spectra and used for size and shape analysis. The area filled by the binary image provided an accurate measurement of per cent oxide minerals in the sample. Although armalcolite and chromite are included with ilmenite in this process, all three minerals are potential oxygen sources. Ilmenite predominates among these oxide minerals, and we use *ilmenite* throughout for convenience in describing the oxide mineral group.

Between 250 and 800 ilmenite grains were analyzed per sample with the sizing program. This program provided measurements for each ilmenite of area, perimeter, average diameter, length, width, shape factor [$\text{perimeter}^2/(4\pi \text{ area})$], aspect ratio (length/width), and orientation. These data were transferred to a computer file from which the graphs in this paper were prepared.

Ilmenite in High-Ti lavas

Width. The best basalt textures to seek for ilmenite beneficiation are coarse-grained, with ilmenite grains having low aspect ratios and low shape factors. Width is the most significant of the measurements, for it controls the size to which the rock must be crushed to extract clean ilmenite grains.

Widths of ilmenite phenocrysts ranged from <10-850 μm (Fig. 1). *Mean* widths in the samples studied ranged from 17-131 μm (Fig. 2) and correlate reasonably well with the ilmenite abundance in the samples (typically 9.0-19.4%).

Based upon width information alone, ilmenites in high-Ti lavas fall into three general categories (Fig. 1 and Table 1): (1) fine-grained, with mean widths of 17—35 μm, which plot as steep curves having standard deviations of 19—68 μm, (2) medium-grained, with mean widths of 40-56 μm and standard deviations of 31-108 μm, and (3) coarse-grained, with mean widths ranging from 105-131 μm and standard deviations of 108-142 μm.

Shape Factor and Aspect Ratio. In published descriptions of lunar ilmenites, shape descriptions are subjective, and include the terms "blocky," "skeletal," "acicular," "laths," "tabular," and "feathery," but it is difficult to quantitatively compare grains or crystals using these terms. The sizing program used here determines a shape factor, which is [perimeter^2/(4π area)] and an aspect ratio (length/width) for each grain. The shape factor is 1.00 for any section through a sphere, and is 1.27 for a centered section through a cube. Thus the shape factor correlates with an increase in surface area over volume.

Ilmenite shape factors for simple, blocky ilmenites show a positive correlation with increasing grain size, for individual samples (Figs. 3a, 4a & b). Ilmenites with quench crystal textures (dendritic, skeletal) (Figs. 3a, 4c &d), with grain size of less than 200μm, show a similar but less pronounced correlation. However, when comparing mean shape factors between samples, there is no correlation between mean width or area with shape factors.

Aspect ratios measured within individual lava samples are mostly less than 3.0 for blocky, coarse-grained ilmenites, but show a broad variation (1.0 to 9.5) for dendritic or skeletal ilmenites. Between samples there is a correlation of increasing mean aspect ratio with decreasing mean width (Table 2).

Ilmenite in Pyroclasts and Regolith Breccias

Nearly all ilmenites in the partly crystalline orange glass droplets from the Apollo 17 landing site are fine-grained and have dendritic shapes (Heiken and McKay, 1977). In the sample examined here (72504), which contains 11.5% ilmenite, the mean area of ilmenite grains is 1.5 μm^2, the mean width is 0.9 μm^2, and the mean aspect ratio is 2.47. Grain widths range from 0.2 to 7.5 μm (Fig. 5).

Ilmenite grains in regolith breccias are coarser-grained than those in orange glass droplets, but are still not an important component. Typical is sample 10046, in which the range of ilmenite widths is 2 to 38 μm, but the volume of ilmenite in the sample is only 3.5 % (Fig. 6).

Ilmenite in the Lunar Regolith

The first evaluations of ilmenite as a resource were made for ilmenite from the lunar regolith (McKay and Williams, 1979). They concluded that regoliths developed on high-Ti mare basalts may have as much as 5 % ilmenite. A soil collected from near station 1, Apollo 17 site (71060) is one of the most ilmenite-rich soils in the sample collection. It is an immature soil (I_s/FeO = 14; M_z = 114 μm;

27% agglutinates) that could be processed for free ilmenite grains. Even so, at grain sizes of <250 µm, free ilmenite makes up <2% of the sample. In the >250 µm size fraction, all ilmenite is bound up in basalt clasts, which must be crushed for processing.

The deepest sampled section of lunar regolith was collected at the Apollo 17 site (samples 70001 to 70009). Variation of opaque minerals (mostly ilmenite) with depth in this regolith corresponds with variations in maturity; immature soils contain the most ilmenite, whereas mature soils do not—the ilmenites are mostly bound up in agglutinates. Variation of free opaque oxides with depth ranges from zero to 4.8 %. The highest abundances are in the less-than-0.2-mm fraction of immature soils (Fig. 7; data from Vaniman and Papike, 1977; Vaniman et al., 1979; Taylor et al., 1977; Taylor et al., 1979).

Beneficiation of Ilmenite from Lunar Rocks and Regolith

The concentration of ilmenite from regolith, regolith breccia or a high-Ti basaltic lava requires sizing to avoid polycrystalline fragments. Key factors in the most efficient beneficiation of ilmenite are grain size, grain width, and shape factors. Figure 8 is a plot constructed by multiplying the Y-axis (Fig. 1; cumulative curve of total ilmenite versus width) by the modal percentage of ilmenite for each sample and inverting the axis. This figure can be used to determine the volume percent of free ilmenite that can be extracted by crushing to different grain sizes. For example, if a coarse-grained high-Ti basalt, containing 19.5% blocky ilmenites (Fig. 3a), is crushed to 200 μm then 13% relatively clean single ilmenite crystals can be freed from the lavas.

If the purpose of the beneficiation is only to extract ilmenite, then the ideal rocks to mine are coarse-grained high-Ti basalts. As can be seen from Fig. 8, little or no free ilmenite can be extracted from lavas with quench-texture and finer-grained ilmenites (such as the one in Fig. 4d), from regolith breccias, or from Apollo-17 orange glasses; in fact, micro-processing would be required to extract even 1% of clean ilmenite from these deposits, because they would need to be crushed to a grain size of <20 μm. In regolith breccias similar to the high-Ti mare soils, very little or no clean ilmenite will be obtained (Fig. 8).

Regoliths as sources of ilmenite have both advantages and disadvantages. If the regolith is to serve multiple uses, including a source of solar-wind-implanted hydrogen, free iron, and feedstock for glass making, then the 1% ilmenite that might be obtained by quarrying and beneficiation is a bonus. If the goal is to obtain the maximum amount of clean ilmenite with the minimum amount of energy needed to crush and separate, then high-Ti mare lavas are the obvious choice for quarrying.

Where to Look for Ilmenite Resources

Textures of lunar high-Ti basalts have been used to interpret the spectrum of cooling rates for these lavas, to evaluate the role of crystal settling within the lava flows, and to estimate the flow thicknesses. From these studies and from photogeologic interpretations of the lunar surface, we have constructed a hypothetical lunar high-Ti lava flow (Fig. 9).

Usselman et al. (1975) determined that the ilmenite shapes represented in the Apollo 17 lava samples indicate cooling rates ranging from 210°C/hr (curved dendrites) to 0.1°C/hr (subhedral, equant and tabular). This range in cooling rates can be explained only if flows are a few m to a few tens of m thick. The lavas sampled at the Apollo 17 landing site are not like those of pooled lavas or very thick flows.

Although not high-Ti basalts, the extensive lava flows of Mare Imbrium have lobate flow scarps with heights ranging from 10 m to 63 m (average=35 m; Schaber, 1973; Gifford and El Baz, 1978). These thicknesses seem to be fairly constant over large distances (as much as 1200 km). Brett (1975) estimated, on the basis of petrologic models, that basalt samples from the Apollo 11, 12, and 15 sites were at least 10 m thick.

In the walls of Hadley Rille, Apollo 15 site, at least three basalt flow units were observed in the upper 60 m. Supporting evidence from photogeologic studies of this site demonstrate that most flows in this area are about 20 m thick (Howard et al., 1973; Gifford and El Baz, 1978; Spudis and Ryder, 1985).

Physical variations within a terrestrial plateau basalt flow, which may be similar in many ways to the mare flows of the Moon show that the lower half of these flows usually consists of massive, dense holocrystalline basalt, whereas the upper half consists of massive basalt with quench textures, grading up into a vesicular top broken by cooling joints (Lutton, 1969; Swanson and Wright, 1978; Arndt et al., 1977).

Our model Apollo 17 high-Ti lava flow consists of a dense, holocrystalline lower half (~12 m thick); this part of the flow contains coarse-grained ilmenites, which may have also been concentrated somewhat by crystal settling. The upper half of the flow contains mostly quench texture ilmenites; complex fine-grained phases that would be difficult to extract by crushing.

The thickness of holocrystalline, coarse-grained basalt in our hypothetical lava flow may be even greater. The immature ejecta in the upper part of the Apollo 17 drillcore, which is inferred to be from Camelot Crater (600-m-diameter crater on the valley floor), consists of 80% equigranular basalt clasts (with coarse, tabular ilmenite) and 20% finer-grained basalt clasts with dendritic or skeletal ilmenite clasts (Vaniman and Papike, 1977). If these clasts represent the ratio of basalt textures represented in flows penetrated by Camelot Crater, then our coarse-grained ilmenite basalt resource is larger than predicted in our model.

Siting a mine for ilmenite would require information on regolith and lava flow thicknesses and coring of the flow(s) to verify the textural models. If these flows have been penetrated by impacts, then the quarry should be sited on the crater rims, where the basalt has already been partly crushed and may be collected as blocky material around the crater rim.

In early analyses, it appeared that the lunar regolith developed on high-Ti basaltic lava flows and lunar pyroclastic deposits are good sources for ilmenite. This is not the case; regolith and pyroclasts may be excellent resources for other purposes, but are not good sources of free or "clean" ilmenite grains.

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Figure Captions

Figure 1. Cumulative curves of fraction of total ilmenite vs. width of ilmenite grains. See Table 1 for sample listing and mean widths for each sample. The curves shown are from fine-grained rocks with quench-texture ilmenites on the left to coarse-grained rocks with equant ilmenites on the right.

Figure 2. Per cent ilmenite within each sample of high-Ti basalt vs. median width of ilmenite.

Figure 3.

a. Ilmenite shape factors, plotted against average ilmenite diameter. Quickly cooled rocks, with small, dendritic ilmenite grains show a large variation in shape factor within a small size range, whereas coarser-grained rocks show a correlation between size and shape factor.

b. Ilmenite aspect ratio vs. area of ilmenites (in a thin section). As for shape factor, there is a broad range of aspect ratios (from 1 to 9.5) for rocks with a dendritic texture; coarser-grained rocks contain ilmenites with aspect ratios of only 1 to 3.5.

Figure 4. Drawings of ilmenites in samples of high-Ti basaltic lavas.

a. 70017,117; mean width (mw)= 105 µm, mean shape factor (sf)=3.26, and mean aspect ratio (ar)=1.86.

b. 74275,84; mw= 28 µm, sf=4.32, and ar=2.22.

c. 75035,78; mw= 60 µm, sf=3.06 and ar=2.37.

d. 70215, 89; mw=19 µm, sf=4.1, and ar=2.57.

Figure 5. Cumulative distribution of ilmenite widths, for a sample of Apollo l7 orange glass sample 72504. Pay attention to the x-axis; the ilmenites are very fine-grained.

Figure 6. Cumulative distribution of ilmenite widths for samples of Apollo 17 orange glass sample 72504 (11.5% ilmenite) and Apollo 11 regolith breccia 10046 (3.5% ilmenite).

Figure 7. Variation, with depth of opaque oxides within the Apollo 17 regolith drillcore (70001-70009). Data from Vaniman and Papike (1977), Vaniman et al. (1979), Taylor et al. (1977), and Taylor et al. (1979).

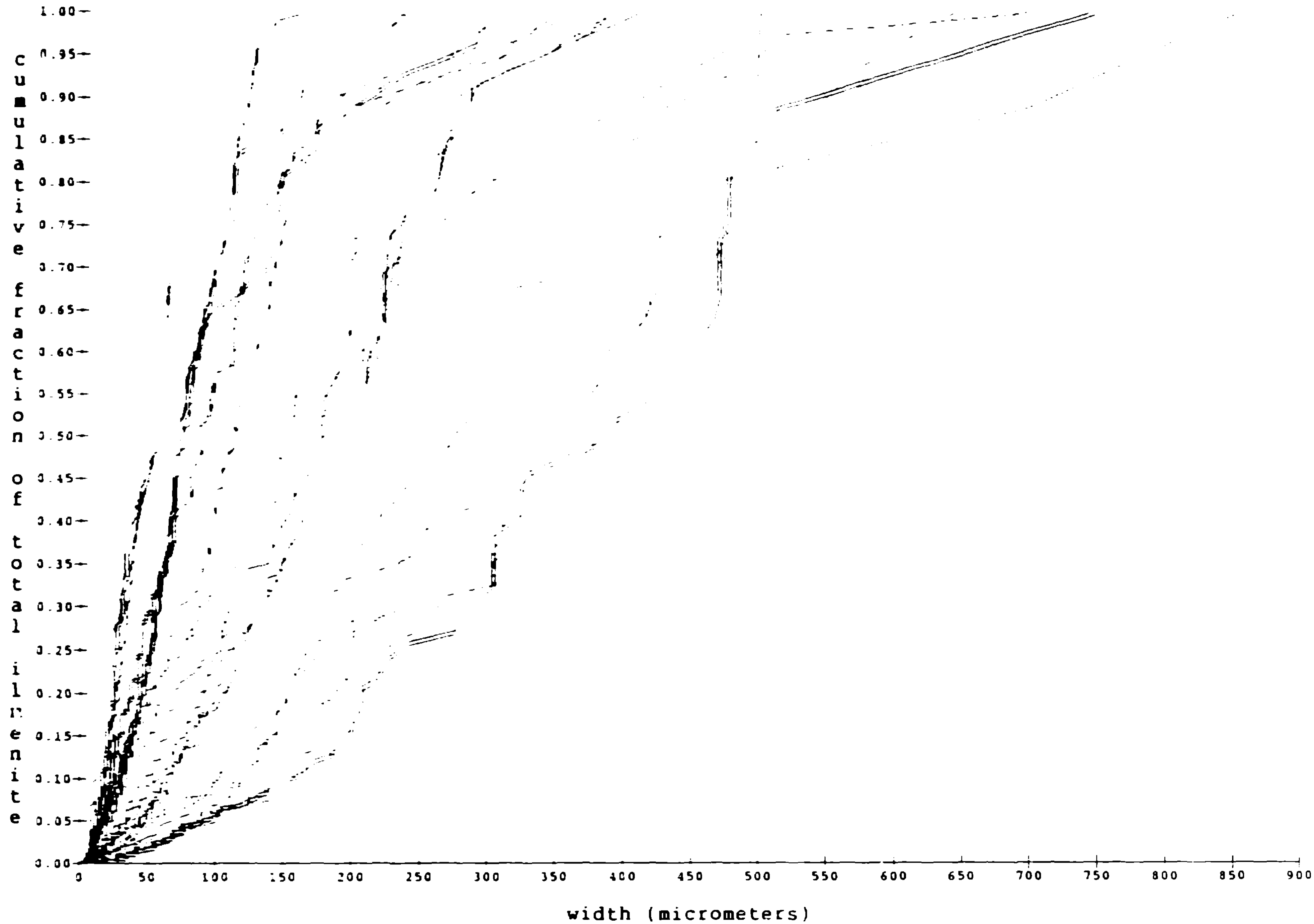
Figure 8. The volume per cent of "clean" ilmenite obtained by crushing vs. minimum crush size. This graph was created by combining cumulative fraction of total ilmenite (Fig. 1) with the per cent ilmenite in each sample. The obvious choices as an ilmenite resource are the coarse-grained, high-Ti lavas (curves on the right). To obtain "clean" ilmenite from either regolith breccias or orange glass deposits would require crushing to less than 10 μm to obtain less than 2%.

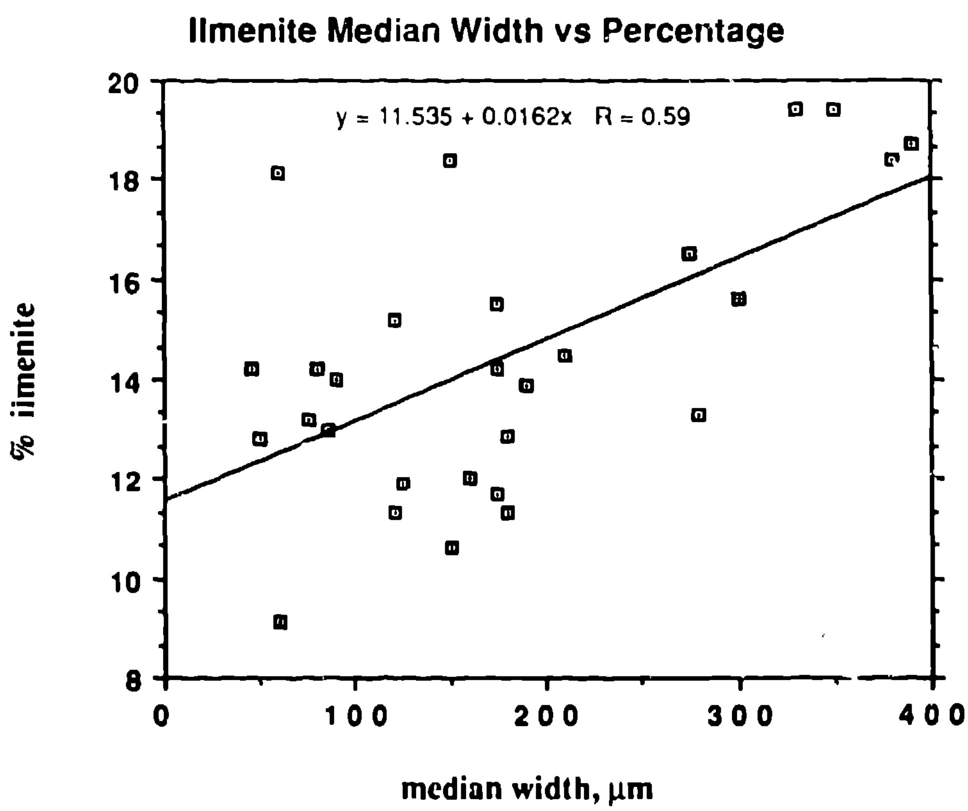
Figure 9. Model high-Ti basaltic lava flow. The source for coarse-grained, blocky ilmenite crystals is within the lower half of this flow. Quarrying is possible, but if flows such as these are penetrated by impact craters, then the coarse-grained ejecta around crater rims would be the ideal source for ilmenite-rich lavas that have already been partly comminuted.

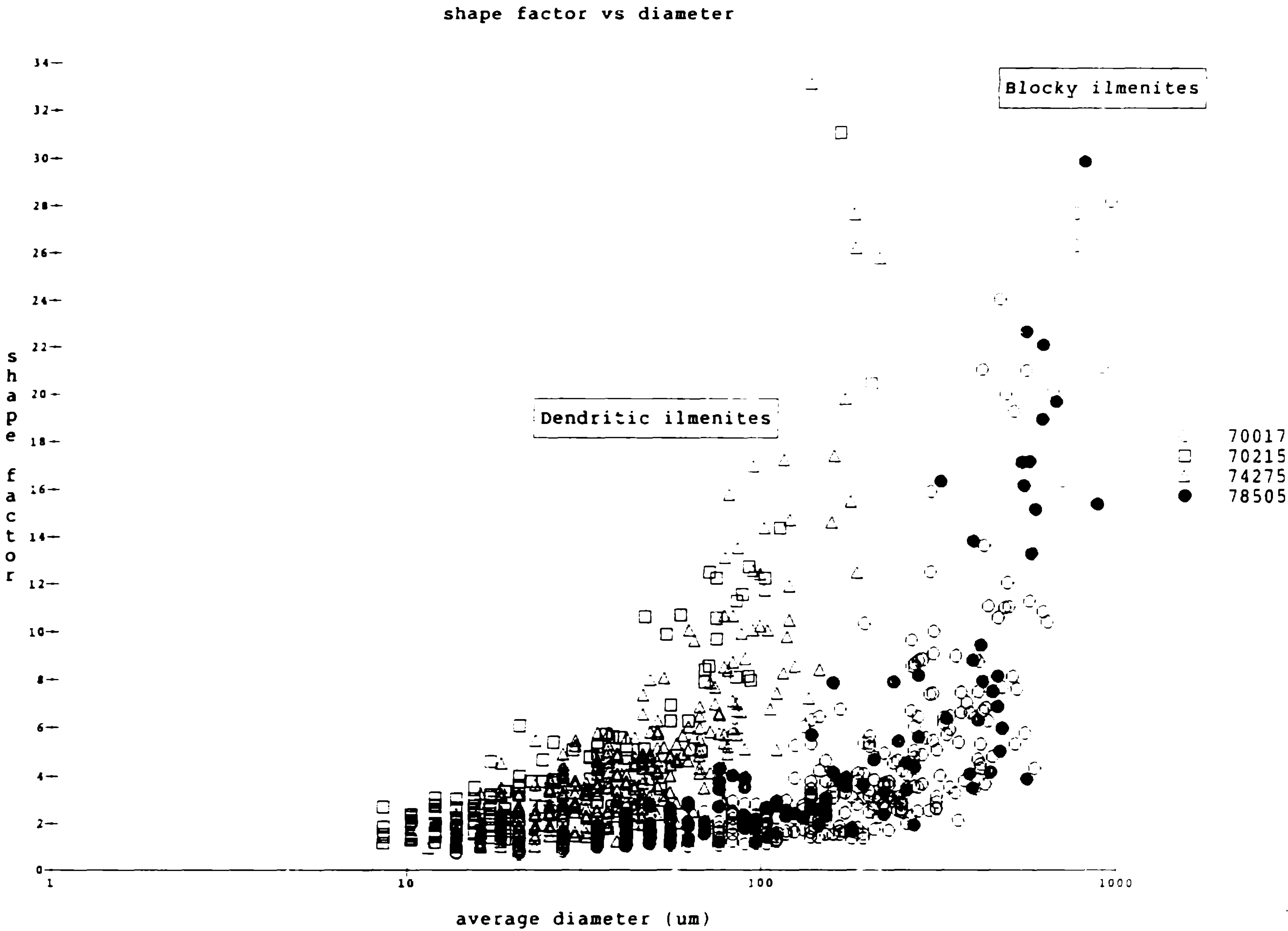
Tables.

Table 1. Samples used in this study, showing means, skewness and standard deviation for ilmenite widths, and means and standard deviation for ilmenite shape factors and aspect ratios.

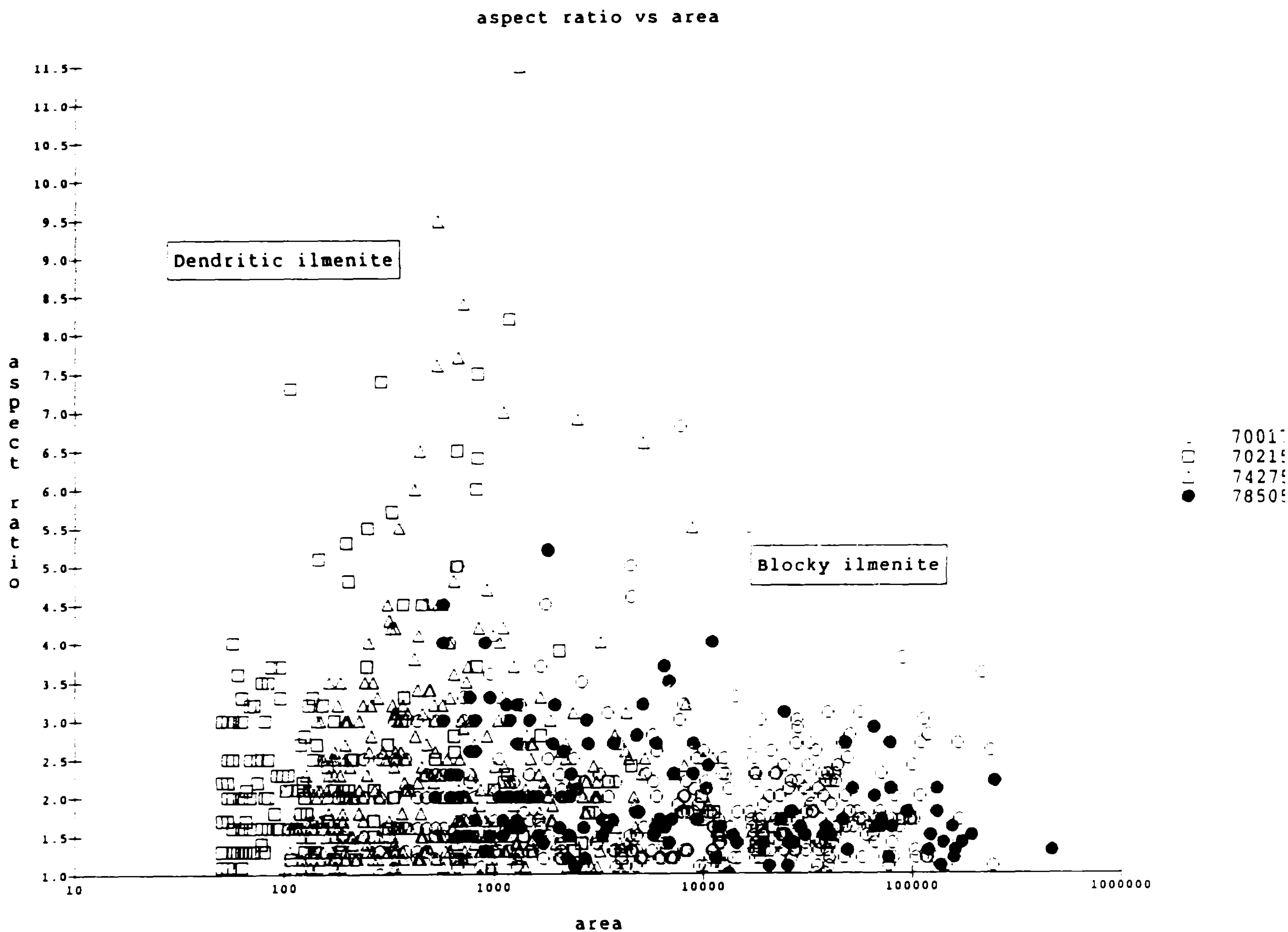
ilmenite width distributions

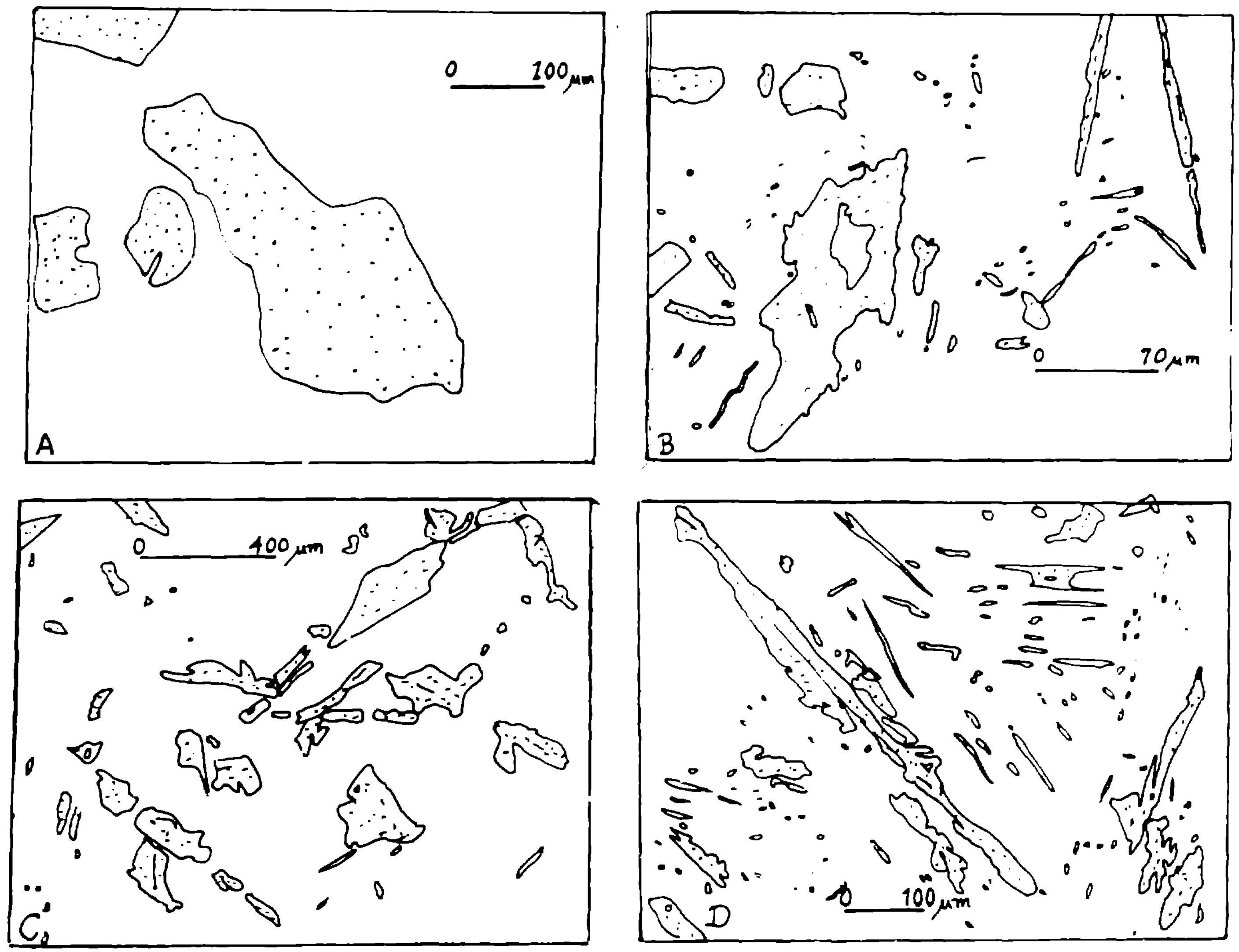


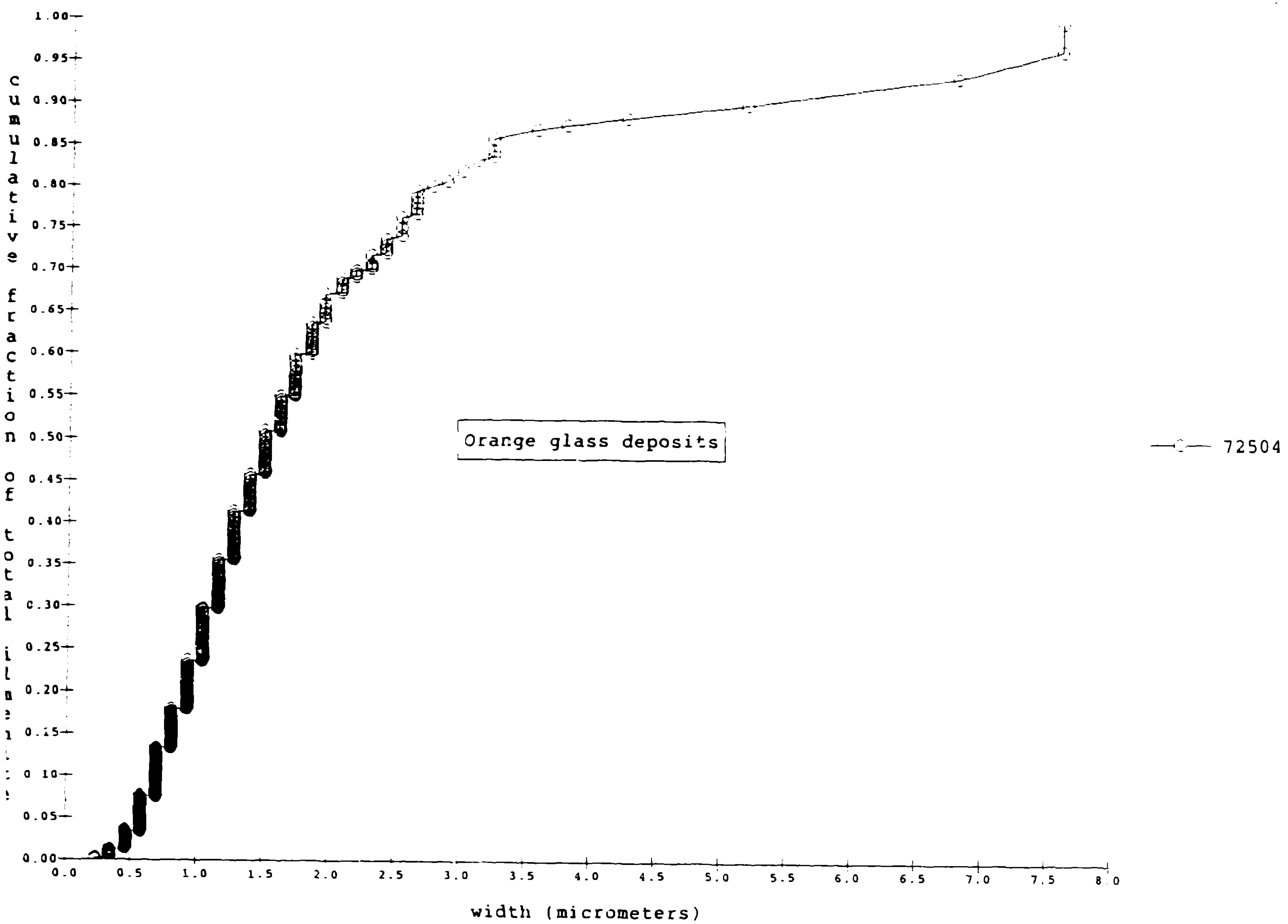

Ilmenite Median Width vs Percentage
y = 11.535 + 0.0162x R = 0.59
% ilmenite
median width, µm
20
18
16
14
12
10
8
0
100
200
300
400



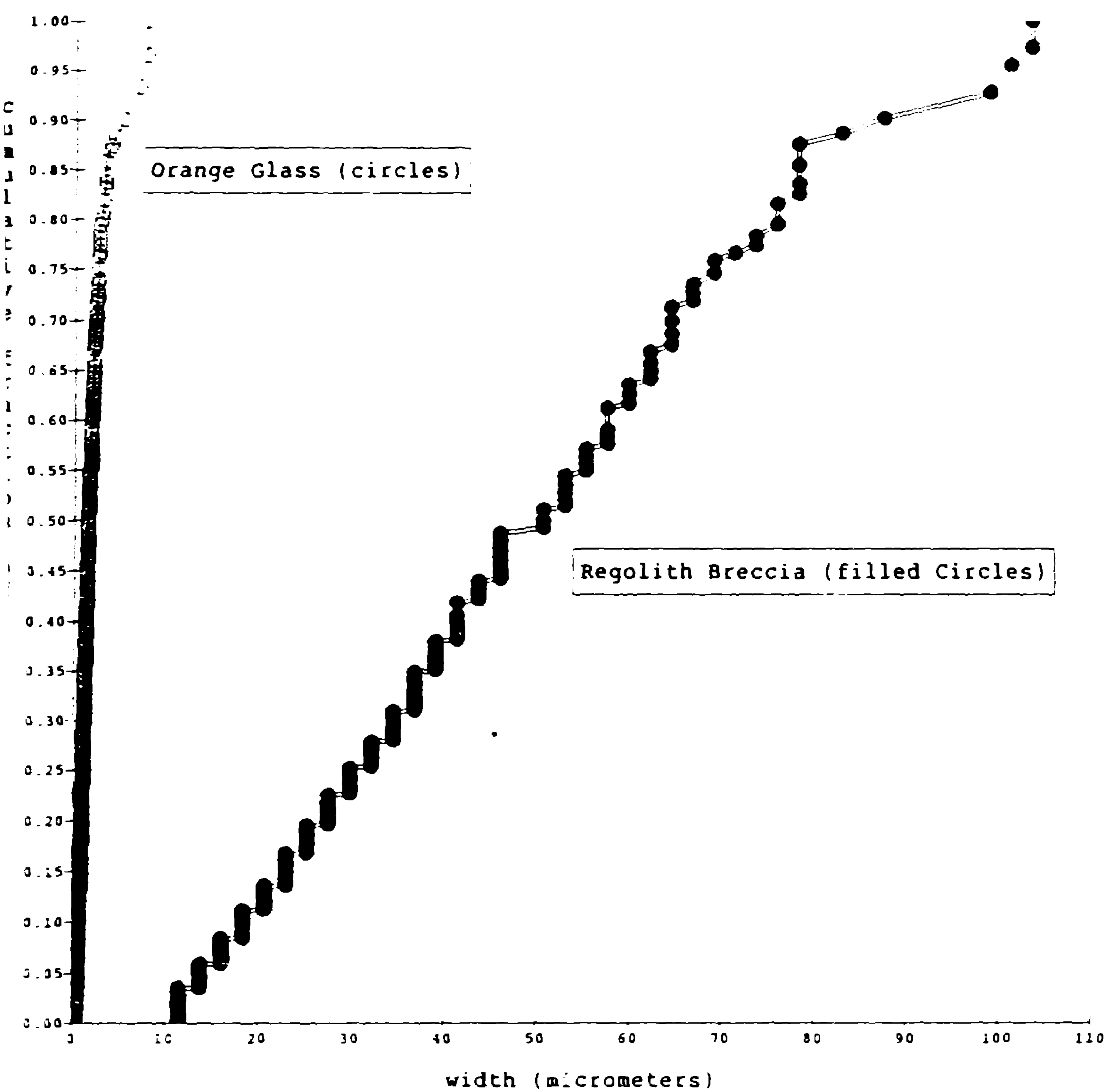
shape factor vs diameter
Blocky ilmenites
Dendritic ilmenites
shape factor
average diameter (um)
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
32
34
1
10
100
1000
70017
70215
74275
78505


aspect ratio vs area
aspect ratio
area
Dendritic ilmenite
Blocky ilmenite
7001
7021
7427
7850
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
10
100
1000
10000
100000
1000000


0 100 μm
A
0 70 μm
B
0 400 μm
C
0 100 μm
D

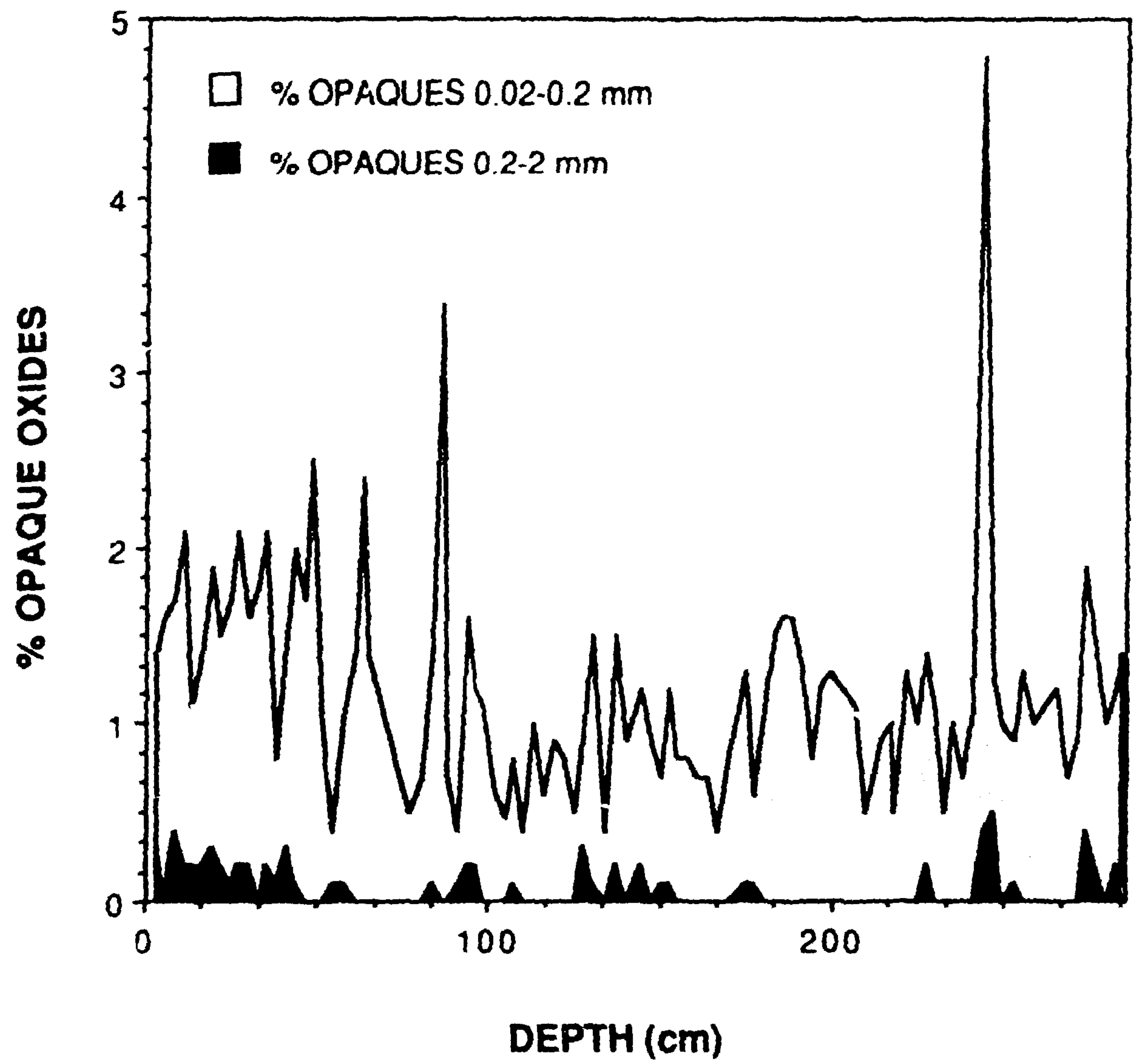


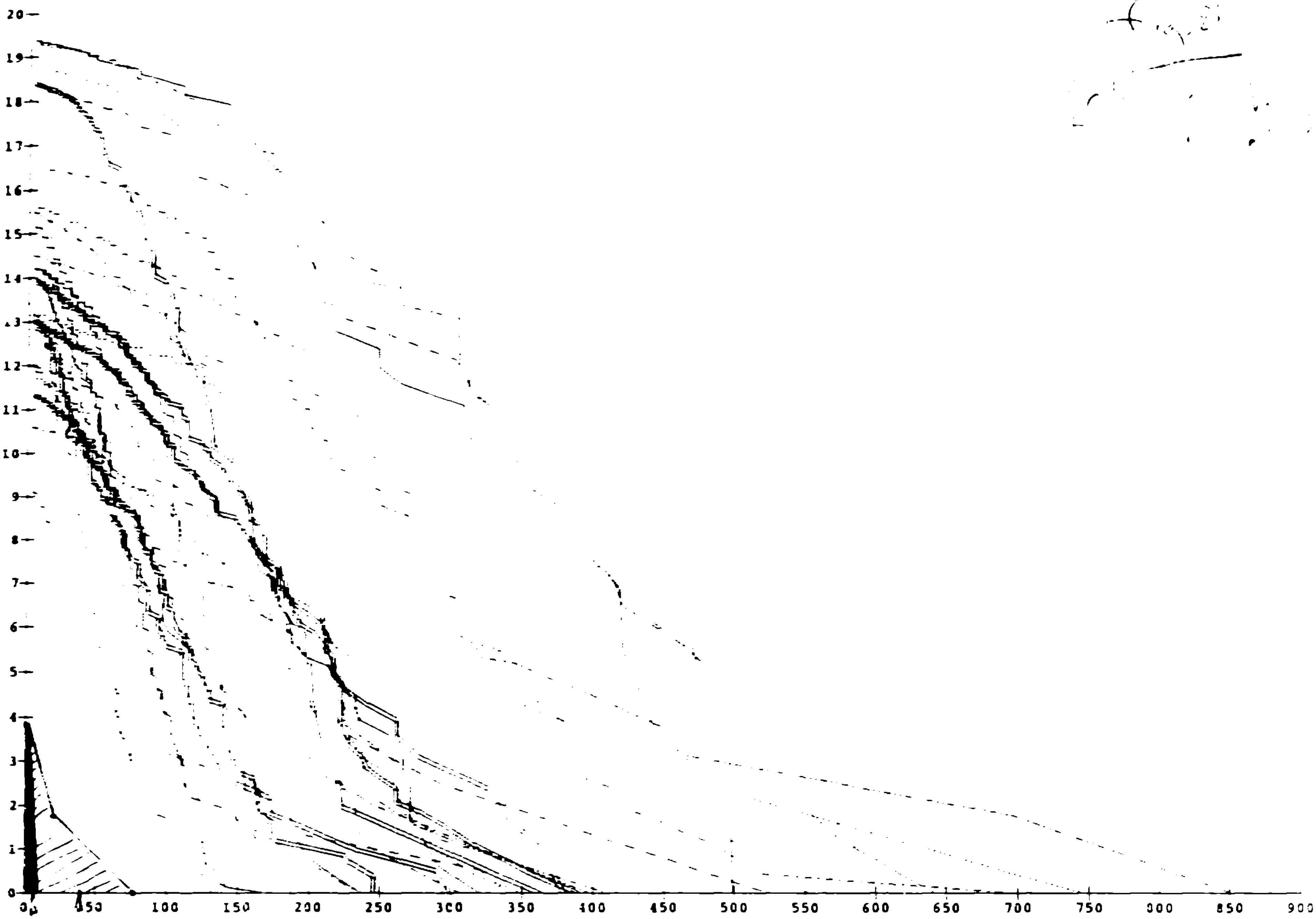
cumulative fraction of total ilmenite
width (micrometers)
Orange glass deposits
72504



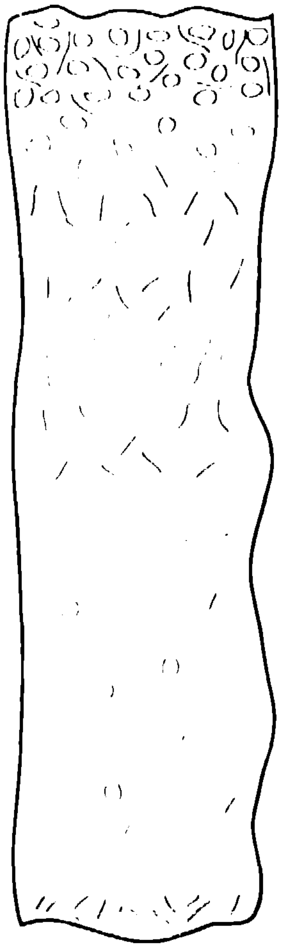
Orange Glass (circles)
Regolith Breccia (filled Circles)
Cumulative Fraction
width (micrometers)
0.00
0.05
0.10
0.15
0.20
0.25
0.30
0.35
0.40
0.45
0.50
0.55
0.60
0.65
0.70
0.75
0.80
0.85
0.90
0.95
1.00
0
10
20
30
40
50
60
70
80
90
100
110

Variation, With Depth, of Opaque Oxides Within the Apollo 17 Regolith Drillcore




minimum crush size (micrometers)

25-m Thick, High-Ti Lava Flow



vesicular, fractured zone (vitric, dendritic crystals) ~3m (12%)

quench textures (dendrites, acicular skeletons) ~10m (40%)

dense, holocrystalline (subhedral, tabular) ~12m (48%)

quench textures at base (?)

(Ratio of holocrystalline, basalts with tabular ilmenite to basalts having dendrite or acicular skeletal ilmenites is ~8:2; Apollo 17 drill core)

Sample No.	Mean width, µm	Stddev. width	Skewness, width	Mean shape factor	Stddev. shape factor	Mean aspect ratio	Stddev. aspect ratio	Ilmenite per cent
10017	41	31	1.15	3.09	2.44	1.98	0.79	14.5
10020	29	33	3.69	4.23	5.48	2.71	1.34	17.1
10050	33	68	4.2	1.98	4.73	2.02	0.6	13.3
10057	28	21	1.9	4.39	3.09	2.37	1.07	15.7
70017	105	108	1.64	3.26	3.92	1.86	0.69	16.5
70035	53	101	3.01	2.43	5	2.06	0.77	15
70135	55	98	2.57	6.13	17.04	2.09	0.96	19.4
70185	53	68	2.16	3.75	5.32	1.96	0.86	13
70215	17	19	3.66	4.1	3.93	2.57	1.41	20
70255	22	29	2.75	2.94	4.13	2.33	1.55	13.3
70275	21	24	4.81	3.67	3.65	2.65	1.32	9.1
70315	33	56	3.01	3.04	5.5	2.25	0.93	15.5
71035	49	59	2.17	2.98	3.68	2.11	1.01	14.2
71055	47	64	2.55	3.39	5.5	2.26	1.51	13.9
71135	48	59	2.43	3.43	4.78	2.31	0.99	11.7
71136	28	36	3.72	2.37	3.37	2.17	1.05	11.9
71155	36	45	3.08	4.42	6.94	2.35	1.26	11.3
71175	56	92	3.92	3.12	5.54	2.08	0.83	15.6
72155	26	34	2.92	2.42	3.67	2.28	0.97	15.2
74255	44	44	1.88	3.12	3.09	2.09	3.09	11.3
74275	28	18	1.61	4.32	3.97	2.22	1.28	14.2
75035	33	60	4.06	3.06	1.08	2.37	1.08	12
75055	36	57	2.84	2.95	4.36	2.06	0.83	10.7
75075	35	65	2.75	2.86	5.4	2.14	0.94	14.5
76136	26	30	2.66	4.85	6.97	2.37	1.02	14
78135	53	63	1.94	7.54	10.56	2.14	0.96	18.4
78505	131	142	1.73	4.56	5.17	2.04	0.77	18.7
78506	31	78	4.54	3.43	12.1	2.17	0.89	18.5

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